

**A SOFT, STRONG, ABSORBENT MATERIAL
FOR USE IN ABSORBENT ARTICLES**

Cross-Reference to Related Application

5 This application is a continuation-in-part application of United States Patent Application Serial No. 08/948,987 filed October 10, 1997, which itself is a continuation-in-part application of United States Patent Application Serial No. 08/784,536 filed January 17, 1997. The disclosures of both applications are incorporated herein by reference.

10

Technical Field of the Invention

 This invention relates to absorbent materials to be used as absorbent cores in articles such as disposable diapers, feminine hygiene products and incontinence devices. More particularly, the present invention relates to absorbent materials that
15 are high density, strong, soft materials with superior absorption properties.

Background of the Invention

 Disposable absorbent articles, such as diapers, feminine hygiene products, adult incontinence devices and the like have found widespread acceptance. To
20 function efficiently, such absorbent articles must quickly absorb body fluids, distribute those fluids within and throughout the absorbent article and be capable of retaining those body fluids with sufficient energy to dry the surface when placed under loads. In addition, the absorbent article need be sufficiently soft and flexible so as to comfortably conform to body surfaces and provide close fit for lower
25 leakage.

 While the design of individual absorbent articles varies depending upon use, there are certain elements or components common to such articles. The absorbent article contains a liquid pervious top sheet or facing layer, which facing layer is designed to be in contact with a body surface. The facing layer is made of a
30 material that allows for the unimpeded transfer of fluid from the body into the core of the article. The facing layer should not absorb fluid *per se* and, thus, should

remain dry. The article further contains a liquid impervious back sheet or backing layer disposed on the outer surface of the article and which layer is designed to prevent the leakage of fluid out of the article.

5 Disposed between the facing layer and backing layer is an absorbent member referred to in the art as an absorbent core. The function of the absorbent core is to absorb and retain body fluids entering the absorbent article through the facing layer. Because the origin of body fluids is often localized, it is necessary to provide a means for distributing fluid throughout the dimensions of the absorbent core to make full use of all the available absorbent material. This is typically
10 accomplished either by providing a distribution member disposed between the facing layer and absorbent core and/or altering the composition of the absorbent core *per se*.

15 Fluid can be distributed to different portions of the absorbent core by means of a transfer or acquisition layer disposed between the facing layer and core. Because of the proximity of such an acquisition layer to the body surface of the wearer, the acquisition layer should not be formed from material that retains large amounts of fluid. The purpose of the acquisition layer is to provide for rapid transfer and distribution of fluid to the absorbent core while minimizing spread of the fluid in this layer.

20 The absorbent core is typically formulated of a cellulosic wood fiber matrix or pulp, which pulp is capable of absorbing large quantities of fluid. Absorbent cores can be designed in a variety of ways to enhance fluid absorption and retention properties. By way of example, the fluid retention characteristics of absorbent cores can be greatly enhanced by disposing superabsorbent materials in amongst fibers of
25 the wood pulp. Superabsorbent materials are well known in the art as substantially water-insoluble, absorbent polymeric compositions that are capable of absorbing large amounts of fluid in relation to their weight and forming hydrogels upon such absorption. Absorbent articles containing blends or mixtures of pulp and superabsorbents are known in the art.

30 The distribution of superabsorbents within an absorbent core can be uniform or non-uniform. By way of example, that portion of an absorbent core proximate to

the backing layer (farthest away from the wearer) can be formulated to contain higher levels of superabsorbent than those portions of the core proximate the facing or acquisition layer. By way of further example, that portion of the core closest to the site of fluid entry (e.g., acquisition zone) can be formulated to transport (wick) fluid into surrounding portions of the core (e.g., storage zone).

In addition to blending pulp with superabsorbent material, a variety of other means for improving the characteristics of pulp have been described. For example, pulp boards can be more easily defiberized by using chemical debonding agents (see, e.g., U.S. Patent No. 3,930,933). In addition, cellulose fibers of wood pulp can be flash-dried prior to incorporation into a composite web absorbent material (see, e.g., U.K. Patent Application GB 2272916A published on June 1, 1994). Still further, the individualized cellulosic fibers of wood pulp can be cross-linked (see, e.g., U.S. Patent Nos. 4,822,453; 4,888,093; 5,190,563; and 5,252,275). All of these expedients have the disadvantage of requiring the wood pulp manufacturer to perform time-intensive, expensive procedures during the wood pulp preparation steps. Thus, use of these steps results in substantial increases in the cost of wood pulp.

Although all of the above treatment steps have been reported to improve the absorption characteristics of pulp for use as absorbent cores, there are certain disadvantages associated with such treatments. By way of example, the manufacturer of the end use absorbent article (e.g. feminine hygiene product or diaper) must fluff the fibers in the wood pulp so as to detach the individual fibers bound in that pulp. Typically, pulp has a low moisture content, which results in the individual fibers being relatively brittle resulting in fine dust due to fiber breakage during fluffing operation. If the pulp manufacturer performs such fluffing prior to shipment to the absorbent article maker, the transportation costs of the pulp are increased. At least one pulp manufacturer has attempted to solve this problem by producing flash-dried pulp without chemical bonding agents in a narrow range of basis weights in pulp density (see U.S. Patent No. 5,262,005). However, even with this process, the manufacturer of the absorbent article must still process the pulp after purchase.

There have been numerous attempts by the manufacturers of absorbent materials to produce highly absorbent, strong, soft core materials. United States Patent No. 4,610,678 discloses an air-laid material containing hydrophilic fibers and superabsorbent material, wherein the material is air-laid in a dry state and compacted without the use of any added binding agents. Such material, however, has low integrity and suffers from shake-out or loss of substantial amounts of superabsorbent material. United States Patent No. 5,516,569 discloses that superabsorbent material shake-out can be reduced in air-laid absorbents by adding significant amounts of water to material during the air-laying process. The resultant material, however, is stiff, of low density and has a high water content (> about 15 weight percent). United States Patent No 5,547,541 discloses that high density air-laid materials containing hydrophilic fibers and superabsorbent material can be made by adding densifying agents to the material. The use of such agents, however, increases the production cost of the material.

United States Patent No. 5,562,645 discloses low density (density less than 0.25 g/cc) absorbent materials. The use of such low density, bulky materials increases the cost of transportation and handling. United States Patent No. 5,635,239 discloses an absorbent material that contains two complex forming agents that interact when wetted to form a complex. The complex forming agents are polymeric olefins. European Patent Application No. EP 0763364 A2 discloses absorbent material that contains cationic and anionic binders that serve to hold the superabsorbent material within the material. The use of such agents and binders increase the cost of making the absorbent material and pose a potential environmental hazard.

There continues to be a need in the art, therefore, for a material that satisfies the absorbency, strength and softness requirements needed for use as absorbent core in disposable absorbent articles and which simultaneously provides time and cost savings to both the pulp manufacturer and the manufacturer of the absorbent article.

Brief Summary of the Invention

In one aspect, the present invention provides an absorbent material having a basis weight of from about 200 g/cm² to about 400 g/cm², a density of from about 0.35 g/cc to about 0.40 g/cc and a ratio of Gurley Stiffness (mg) to density (g/cc) of less than about 3700. The material is airlaid as a bottom layer of pulp, a middle layer of pulp and superabsorbent material disposed in amongst the pulp, and a top layer of pulp. The pulp preferably has a Kappa value of less than about 100. In one embodiment, the absorbent material includes from about 40 weight percent to about 90 weight percent cellulosic fibers and from about 10 weight percent to about 60 weight percent superabsorbent material. Such absorbent material has a water content of less than about 10 weight percent, a density of greater than about 0.25 g/cc, a ratio of Gurley Stiffness (mg) to density (g/cc) of less than about 3700 and a pad integrity of greater than about 12 Newtons.

In another embodiment, the absorbent material includes from about 40 weight percent to about 90 weight percent cellulosic fibers and from about 10 weight percent to about 60 weight percent superabsorbent material. Such absorbent material has a water content of less than about 10 weight percent, a density of greater than about 0.25 g/cc, a ratio of Gurley Stiffness (mg) to density (g/cc) of less than about 3700 and retains more than about 85 weight percent of superabsorbent material after shaking for 10 minutes.

In yet another embodiment, the absorbent material includes from about 40 weight percent to about 90 weight percent cellulosic fibers and from about 10 weight percent to about 60 weight percent superabsorbent material, a water content of less than about 10 weight percent, a density of greater than about 0.25 g/cc, a ratio of Gurley Stiffness (mg) to density (g/cc) of less than about 3700, a ratio of pad integrity (Newtons) to density (g/cc) of greater than about 25.0 and a 45° wicking distribution at 5 inches of at least 7 grams of saline per gram of absorbent material.

With all embodiments, it is preferred that at least some of the cellulosic fibers have a relative crystallinity of less than about 65 percent.

In another aspect, an absorbent material of this invention has a basis weight of from about 100 g/m² to about 500 g/m² and a density of from about 0.25 g/cc to about 0.50 g/cc. Such material includes a core of cellulosic fibers obtained from pulp having a Kappa value of less than about 100 and a layer of tissue superimposed on an outer surface of the core. At least some of the cellulosic fibers have a relative crystallinity of less than about 65 percent. The core contains from about 40 weight percent to about 100 weight percent cellulosic fibers and from about 0 weight percent to about 60 weight percent superabsorbent material. Preferably, the core contains from about 40 weight percent to about 90 weight percent cellulosic fibers and from about 10 weight percent to about 60 weight percent superabsorbent material. The material has a suppleness of greater than about 0.7 g⁻¹. The tissue of such material is preferably crepe tissue.

In one embodiment, the absorbent material has a density of from about 0.25 to about 0.5 g/cc and a suppleness of greater than about 0.7 g⁻¹. Such material consists essentially of from about 40 weight percent to about 90 weight percent cellulosic fibers at least some of which fibers are obtained from pulp having a Kappa value of less than about 100, wherein at least some of the cellulosic fibers have a relative crystallinity of less than about 65 percent and from about 10 weight percent to about 60 weight percent superabsorbent material.

A material of the present invention has a normalized drying power energy of at least about 6000 ergs/g, a normalized wicking energy of at least about 3000 ergs/g or both a normalized drying power energy of at least about 6000 ergs/g and a normalized wicking energy of at least about 3000 ergs/g.

In another embodiment, an absorbent material of this invention has a density of from about 0.25 g/cc to about 0.5 g/cc, a basis weight of from about 200 g/m² to about 500 g/m², a suppleness of greater than about 0.7 g⁻¹, a normalized drying power energy of at least about 6000 ergs/g and a normalized wicking energy of at least about 3000 ergs/g. Such material consists essentially of from about 60 weight percent to about 90 weight percent cellulosic fibers at least some of which fibers are obtained from pulp having a Kappa value of less than about 100, wherein at least some of the cellulosic fibers have a relative

crystallinity of less than about 60 percent; from about 10 weight percent to about 40 weight percent superabsorbent material; and a layer of tissue comprising from about 3 weight percent to about 20 weight percent of the absorbent material. The tissue is preferably crepe tissue.

5 An absorbent material in accordance with all embodiments is made using cellulosic fibers having a relative crystallinity of preferably less than about 60 percent. More preferably, the cellulosic fibers have a relative crystallinity of less than about 50 percent and, even more preferably a relative crystallinity of less than about 40 percent.

10 At least some of the cellulosic fibers are obtained from pulp having a Kappa value of less than about 75, 50, 25 or 10. More preferably, the Kappa value is less than 5 or 2.5.

15 In one preferred embodiment, at least some of the cellulosic fibers in the material is made by a process that includes the step of treating a liquid suspension of pulp at a temperature of from about 15°C to about 60°C with an aqueous alkali metal salt solution having an alkali metal salt concentration of from about 2 weight percent to about 25 weight percent of said solution for a period of time ranging from about 5 minutes to about 60 minutes. In another embodiment, at least some of the cellulosic fibers have been flash dried.

20 The material of the present invention has superior absorptive properties. The material has a normalized drying power energy of at least 6,000 ergs/g. Preferably, the normalized drying power energy of the material is greater than about 7,000, 8,000, 9,000 or 10,000 ergs/g. More preferably, the normalized drying power is between about 6,000 ergs/g and about 16,000 ergs/g. The material has a
25 normalized wicking energy of at least 3,000 ergs/g. Preferably, the normalized wicking energy is greater than about 3,500, 4,000, 5,000, or 7,500 ergs/g. More preferably, the normalized wicking energy is between about 3,000 ergs/g and about 10,000 ergs/g. In an especially preferred embodiment, the material of this invention has a normalized drying power energy of at least 6,000 ergs/g and a wicking energy
30 of at least about 3,000 ergs/g.

An absorbent material of the present invention is supple. The suppleness, defined as the inverse of stiffness, is greater than about 0.7 g^{-1} . Preferably, the suppleness is greater than about 0.8, 0.9, or 1.0 g^{-1} .

5 The absorbent material most preferably has a suppleness of greater than about 0.7 g^{-1} , a normalized drying power energy of greater than about 6,000 ergs/g and a normalized wicking energy greater than about 3,000 ergs/g.

10 An especially preferred absorbent material of this invention has a density of from about 0.35 g/cc to about 0.45 g/cc, a basis weight of from about 200 g/m^2 to about 500 g/m^2 , a suppleness of greater than about 0.9 g^{-1} , a normalized drying power energy of greater than about 6,000 ergs/g and a normalized wicking energy greater than about 3,000 ergs/g.

15 The present invention still further provides absorbent articles that include an absorbent material of this invention. Preferably, the absorbent article is a diaper, a feminine hygiene product or an incontinence device.

Brief Descriptions of the Drawings

In the drawings, which form a portion of the specification:

20 FIG. 1 is a schematic illustration of means for air-laying absorbent material of the present invention using four air-laying heads followed by means for compacting the air-laid material.

FIG.2 shows three and four strata embodiments of an absorbent material of the present invention for use as an absorbent core in a feminine hygiene product.

FIG. 3 shows a three and four strata embodiments of an absorbent material of the present invention for use in a diaper or incontinence device.

25 FIG. 4 is a schematic illustration of a device for measuring the wicking properties of absorbent material.

FIG. 5 is a representative plot of fluid absorption versus distance obtained in a 45° wicking test.

30 FIG. 6 is a schematic illustration of a device used to measure the drying power of absorbent materials.

FIG. 7 is a representative plot of fluid absorption versus hydrostatic pressure obtained in a drying power test.

FIG. 8 is a representative x-ray diffractogram of cellulosic fibers used in an absorbent material of the present invention.

5 FIG. 9 is a composite x-ray diffractogram of four different cellulosic fibers used in absorbent materials of this invention.

Detailed Description of the Invention

10 The present invention provides novel, absorbent material that is particularly well suited for use as cores in absorbent articles such as diapers, feminine hygiene products, incontinence devices and the like. An absorbent material can also be used as an absorbent core in any device used to absorb body exudates (e.g., urine, breast milk, blood, serum). Thus, the material can be incorporated into breast pads for nursing mother or used as absorbent material in surgical drapes (e.g., towels) or

15 wound dressings. The material of the present invention is a blend or mixture of cellulosic fibers and, optionally, superabsorbent disposed in and amongst fibers of that pulp. A material of this invention has a unique combination of suppleness, strength and absorbency characteristics that makes it particularly suitable for use in absorbent articles. An absorbent material of the present invention can be used

20 directly by a manufacturer of the absorbent article without the need for any additional processing by that manufacturer other than cutting or folding to the desired size and shape for the absorbent article.

 The present invention relates to an absorbent material that is soft, thin, and of high density. Additionally, the material has enhanced absorption properties and

25 firmly entraps superabsorbent material in the fiber network without the use of water, chemicals, binders, adhesives, thermoplastic resins, thermoplastic binder fibers, complex forming materials or the like. The absorbent material has enough integrity (strength) to be processed on conventional disposable product manufacturing equipment without fiber breakage.

30 In one aspect, the present invention provides an absorbent material that contains from about 40 weight percent to about 100 weight percent cellulosic fibers

and from about 0 weight percent to about 60 weight percent superabsorbent material. The absorbent material has a water content of less than about 10 weight percent. As used herein, the phrase "weight percent" means weight of substance per weight of final material as determined under ambient conditions. By way of
5 example, 10 weight percent superabsorbent material means 10 g/m² superabsorbent material per 100 g/m² basis weight of the absorbent material.

Cellulosic fibers that can be used in a material of the present invention are well known in the art and include wood pulp, cotton, flax, and peat moss. The present invention also contemplates that short cut synthetic fibers (up to about 10
10 percent) may be incorporated in the absorbent core. Wood pulp is preferred. Pulps can be obtained from mechanical or chemi-mechanical, sulfite, kraft, pulping reject materials, organic solvent pulps, etc. Both softwood and hardwood species are useful. Softwood pulps are preferred. It is not necessary to treat cellulosic fibers with chemical debonding agents, cross-linking agents and the like for use in the
15 present material.

As set forth above, a preferred cellulosic fiber for use in the present material is wood pulp. Wood pulp prepared using a process that reduces the lignin content of the wood is preferred. Preferably, the lignin content of the pulp is less than about 16 percent. More preferably, the lignin content is less than about 10 percent. Even
20 more preferably, the lignin content is less than about 5 percent. Most preferably, the lignin content is less than about 1 percent. As is well known in the art, lignin content is calculated from the Kappa value of the pulp. The Kappa value is determined using a standard, well known test procedure (TAPPI Test 265-cm 85). The Kappa value of a variety of pulps was measured and the lignin content
25 calculated using the TAPPI Test 265-cm 85. Peat moss was found to have a Kappa value of about 104 and a lignin content of about 13.5 percent. CTMP pulp was found to have a Kappa value of about 123 and a lignin content of about 16 percent. Pulp prepared from softwood using either the kraft or sulfite methods had a Kappa
30 value of about 1.1 and a lignin content of about 0.15 percent. When that latter pulp was treated using a cold caustic extraction method, the Kappa value was found to be about 0.97 and the lignin content about 0.12 percent.

For use in the present invention, cellulosic fibers are preferably obtained from wood pulp having a Kappa value of less than about 100. Even more preferably, the Kappa value is less than about 75, 50, 25 or 10. Most preferably, the Kappa value is less than about 2.5.

5 There are certain other characteristics of wood pulp that make it particularly suitable for use in an absorbent material of the present invention. Cellulose in most wood pulps has a high relative crystallinity (greater than about 65 percent). In a present material, the use of wood pulp with a relative crystallinity of less than about 65 percent is preferred. More preferably, the relative crystallinity is less than about 10 50 percent. Most preferably, the relative crystallinity is less than about 40 percent. Similarly, pulps having an increase fiber curl value are preferred.

Means for treating pulps so as to optimize these characteristics are well known in the art. By way of example, treating wood pulp with liquid ammonia is known to decrease relative crystallinity and to increase the fiber curl value. Flash 15 drying is known to increase the fiber curl value of pulp and to decrease crystallinity. Cold caustic treatment of pulp also increases fiber curl and decreases relative crystallinity. Chemical cross-linking is known to decrease relative crystallinity. It is preferred that the cellulosic fibers used to make the material of this invention are obtained at least in part using cold caustic treatment or flash drying.

20 A description of the cold caustic extraction process can be found in commonly owned United States Patent Application Serial No. 08/370,571, filed on January 18, 1995, which application is a continuation-in-part application of United States Patent Application Serial No. 08/184,377, filed on January 21, 1994. The disclosures of both of these applications are incorporated in their entirety herein by 25 reference.

Briefly, a caustic treatment is typically carried out at a temperature less than about 60°C, but preferably at a temperature less than 50°C, and more preferably at a temperature between about 10°C to 40°C. A preferred alkali metal salt solution is a sodium hydroxide solution newly made up or as a solution by-product in a pulp or 30 paper mill operation, e.g., hemicaustic white liquor, oxidized white liquor and the like. Other alkali metals such as ammonium hydroxide and potassium hydroxide

and the like can be employed. However, from a cost standpoint, the preferable salt is sodium hydroxide. The concentration of alkali metal salts is typically in a range from about 2 to about 25 weight percent of the solution, and preferably from about 6 to about 18 weight percent. Pulps for high rate, fast absorbing applications are preferably treated with alkali metal salt concentrations from about 10 to about 18 weight percent.

As is well known in the art, flash drying is a method for drying pulp in which pulp is partially dewatered, fiberized and fed into a stream of hot air which causes the moisture contained in the pulp to be flashed off. Briefly, the pulp, initially at a consistency of 30-45% (containing 55-70% water), is conveyed directly into a fluffer (e.g., a disk refiner) where mechanical action is used to fiberize (break up and separate) and disperse the fibers for the flash drying system. Once discharged from the fluffer device, the fiberized pulp is fed into a flash drying system. The drying system itself is made up of two stages, each of which consists of two drying towers. The fiber is conveyed through the drying towers by high velocities of hot air. The inlet air temperature for the first stage is approximately 240-260°C while the inlet air temperature for the second stage is approximately 100-120°C. Following each drying stage, the pulp and hot air are then conveyed into a cyclone separator, where the hot air, now containing moisture evaporated from the pulp, is exhausted vertically. Exhaust temperatures for the first stage, in this case, are approximately 100-120°C, and the exhaust temperatures for the second stage are approximately 90-100°C. At the same time, a material-handling fan draws the pulp fibers through the cyclone cone and on to the next part of the system. Finally, following the second stage cyclone separator, the dried pulp is passed through a cooling stage consisting of a cooling fan, conveying ambient air, and a final cooling cyclone separator. The residence time for the entire system, including both drying stages, cyclone separation, and cooling, is approximately 30-60 seconds at the feed rate used (1.5 kg of dry material per minute).

A downside to producing flash dried fiber using the type of system described above is the production of localized fiber bundles in the final product.

Fiber bundles are formed during the fiberization of the pulp by mechanical action within the fluffer device. The system above uses a disk refiner consisting of two grooved, circular plates at a set gap width, in this case 4 mm. One plate is in a fixed position while the other plate is rotated at high speeds. The pulp is fed into the gap between the two plates and the rotation of the plate results in the separation of fibers along the grooves. Unfortunately, as the pulp is fiberized, some of the individual fibers tend to become entangled with one another, forming small bundles consisting of several individual fibers. As these entangled fibers are flash dried and the moisture is removed, the entanglements tighten and harden to form small localized fiber bundles throughout the flash dried pulp. The presence of large numbers of these localized fiber bundles within the final airlaid products produced using the flash dried pulp can result in a reduction of product physical characteristics and performance. The number of localized fiber bundles can be substantially reduced by using cold caustic extracted pulp.

An absorbent material of the present invention can contain any superabsorbent material, which superabsorbent materials are well known in the art. As used herein, the term "superabsorbent material" means a substantially water-insoluble polymeric material capable of absorbing large quantities of fluid in relation to their weight. The superabsorbent material can be in the form of particulate matter, flakes, fibers and the like. Exemplary particulate forms include granules, pulverized particles, spheres, aggregates and agglomerates. Exemplary and preferred superabsorbent materials include salts of crosslinked polyacrylic acid such as sodium polyacrylate. superabsorbent materials are commercially available (e.g., Stockhausen GmbH, Krefeld, Germany).

In accordance with a preferred embodiment, the material contains from about 40 to about 100 weight percent cellulosic fibers and, more preferably from about 60 to about 80 weight percent cellulosic fibers. Such a material preferably contains from about 0 to about 60 weight percent superabsorbent material and, more preferably from about 20 to about 40 weight percent superabsorbent material.

An absorbent material is made using air-laying means well known in the art (See FIG. 1). In accordance with FIG. 1, cellulosic fibers (e.g., pulp) are processed

using a hammer mill to individualize the fibers. The individualized fibers are blended with superabsorbent material granules in a blending system 1 and pneumatically conveyed into a series of forming heads 2. The blending and distribution of absorbent materials can be controlled separately for each forming head. Controlled air circulation and winged agitators in each chamber produce uniform mixture and distribution of pulp and superabsorbent material. The superabsorbent material can be thoroughly and homogeneously blended throughout the web or contained only in specific strata by distributing it to selected forming heads. Fibers (and superabsorbent material) from each forming chamber are deposited by vacuum onto a forming wire 3 thus forming a layered absorbent web. The web is subsequently compressed using heated calendars 4 to achieve desirable density. The densified web is wound into a roll 5 using conventional winding equipment. The forming wire 3 is covered with tissue to reduce the loss of material. The tissue layer is preferably incorporated into the formed material.

Suitable tissue materials for use in absorbent products are well known to one of skill in the art. Exemplary and preferred such tissue is made of bleached wood pulp and has an air permeability of about 273-300 CFM (cubic feet minute). The tensile strength of the tissue is such that it retains integrity during formation and calendering of the absorbent material. Suitable MD and CD tensile strengths, expressed in Newtons/meter, are about 100-130 and 40-60, respectively. Tissue for use in air-laying absorbent materials are commercially available (e.g., Duni AB, Sweden). In a preferred embodiment, the tissue is crepe tissue having a sufficient number of crepes per inch to allow a machine direction elongation of between 15 and 30 percent (as determined by the SCAN P44:81 test method).

An absorbent material of the present invention is of high density and has a density of greater than about 0.25 g/cc. In preferred embodiments, the material has density in the range of from about 0.25 g/cc to about 0.50 g/cc. More preferably, the density is from about 0.30 g/cc to about 0.45 g/cc. Most preferably, the density is from about 0.35 g/cc to about 0.45 g/cc.

Air-laid absorbents are typically produced with a low density. To achieve higher density levels, such as preferred in the material of the present invention, the

air-laid material is compacted using calendars as shown in FIG. 1. Compaction is accomplished using means well known in the art. Typically such compaction is carried out at a temperature of about 100°C and a pressure of about 130 Newtons per millimeter. The upper compaction roll is typically made of steel while the lower compaction roll typically is a flexroll having a hardness of about 85 SH D. It is preferred that both the upper and lower compaction rolls be smooth, although the upper roll can be shallow engraved. As set forth hereinafter in the Examples, use of engraved upper roll may result in formation of a material having slower fluid absorption rates. The preference for calendering with smooth rolls is surprising in light of the teaching of United States Patent No. 5, 516, 569, which teaches that such calendering results in increased Gurley stiffness and damage to the absorbent material.

A high density absorbent material of the present invention that contains superabsorbent material is surprisingly and unexpectedly supple. Such material has a ratio of Gurley stiffness, measured in milligrams (mg) to density, measured in grams per cubic centimeter (g/cc), of less than about 4000. In preferred embodiments, that ratio of Gurley stiffness to density is less than about 3200 and, more preferably, less than about 3000.

Gurley stiffness measures the stiffness of absorbent materials. The greater the value of Gurley stiffness, the more rigid and inflexible the material. The inverse of Gurley stiffness, expressed as inverse grams (g^{-1}), is thus a measure of the softness, bendability and flexibility of absorbent materials. The term "suppleness" is used herein to describe these characteristics of softness, flexibility and bendability. Suppleness is defined and expressed as the inverse of Gurley stiffness and has the units g^{-1} .

As set forth hereinafter in the examples, suppleness was determined on absorbent material of the present invention as well as absorbent core material from two commercially available disposable diapers. The suppleness was determined at a number of different densities. The material of the present invention was substantially and significantly more supple than existing, commercially available material at every density tested. The material of the present invention has a

suppleness of at least 0.7 g^{-1} . Preferably, the suppleness is greater than 0.8, 0.9, or 1.0 g^{-1} .

5 An absorbent material of the present invention is strong in light of its suppleness. Pad integrity is a well known measurement of absorbent material strength. A material of the present invention demonstrates strength (high pad integrity) over a wide range of densities (See the Examples hereinafter). For any given density within the range of 0.25 to 0.50 g/cc, material of the present invention has significantly greater (about 2 to 3 times) pad integrity than does the tested commercially available materials.

10 An absorbent material of this invention can be prepared over a wide range of basis weights without adversely affecting its softness or strength. Thus, the material can have a basis weight in the range of from about 50 g/m^2 to about 700 g/m^2 . In a preferred embodiment, the basis weight ranges from about 100 g/m^2 to about 500 g/m^2 and, more preferably from about 100 g/m^2 to about 250 g/m^2 or
15 from about 350 g/m^2 to about 450 g/m^2 .

In addition to being supple and strong, an absorbent material of the present invention has superior absorptive properties when compared to existing materials. The absorptive properties of materials can be evaluated in a variety of ways. Of particular relevance to manufacturers of absorbent articles is the ability of the
20 material to absorb large quantities of fluid against a load and to distribute that fluid away from the point of fluid entry.

Wicking is the ability of an absorbent material to direct fluid away from the point of fluid entry and distribute that fluid throughout the material. An absorbent material of this invention has surprisingly superior wicking properties when
25 compared to absorbent cores from commercially available absorbent articles (e.g., Huggies® or Pampers® diapers). As described in detail hereinafter in the Examples, the wicking properties of two embodiments of a present invention wicked substantial amounts of fluid over 6 inches from the point of fluid entry. In a 400 g/m^2 basis weight, 20 weight percent superabsorbent material, the 45° wicking
30 distribution at 5 inches was about 8 grams of fluid per gram of material. That same material had a wicking distribution at 7 inches of about 1.7 grams of fluid. Similar

wicking properties were seen in a 600 g/m² basis weight, 40 weight percent absorbent material of the present invention. In contrast, the absorbent core from a Huggies® or Pampers® diaper at similar basis weight and superabsorbent content, had a wicking distribution at 6 inches of less than 1 gram of fluid per gram of material. Neither commercially available diaper core distributed any substantial amounts of fluid beyond 6 inches.

The wicking capability of an absorbent material can be better characterized by expressing the wicking properties over the entire length of a tested sample. As set forth in detail hereinafter in the Examples, by calculating the total amount of fluid absorbed and wicked by a test sample (calculating the areas under a plot of absorbed fluid vs distance), a wicking energy (the capacity of the absorbent material to perform absorptive work) can be calculated. Because absorption is in part a function of superabsorbent material content, that energy can be normalized for superabsorbent material content. The resulting value is referred to herein as "normalized wicking energy" and has the units ergs/g. As set forth in detail hereinafter in Example 6, the normalized wicking energy was determined for absorptive material of the present invention as well as commercially available absorptive material. The data show that an absorptive material of the present invention has a normalized wicking energy of at least about 3,000 ergs/g. More preferably, the normalized wicking energy is greater than about 3,500, 4,000, 5,000, or 7,500 ergs/g. Most preferably, the absorptive material of the present invention has a normalized wicking energy of from about 3,000 to about 10,000 ergs/g. These values can be seen to be significantly greater than the values obtained from other materials.

It is important that an absorbent material designed for use in articles such as diapers, feminine hygiene products and incontinence devices be able to absorb fluid against a hydrostatic pressure gradient. One measure of such an absorptive capacity is drying power, which measures the absorption of fluid against a negative hydrostatic pressure applied to the fluid source. The drying power test is described generally in Burgeni et al., *Textile Research Journal*, 37:362, 1967 and, in detail, hereinafter in Example 7. As was the case for wicking energy, by calculating the

total amount of fluid absorbed during the drying power test (calculating the area under a plot of absorbed fluid vs pressure), the work performed by the tested material can be calculated. As used herein, the phrase "drying power energy" refers to such drying power work. The units of drying power energy are ergs/g. Drying power energy corrected for superabsorbent material content is referred to herein as "normalized drying power energy".

Normalized drying power energy was determined for absorptive material of the present invention as well a number of commercially available materials. The data show that the absorptive material of this invention has a normalized drying power energy of at least 6,000 ergs/g. Preferably, the normalized drying power energy is greater than about 7,000, 8,000, 9,000 or 10,000 ergs/g. Most preferably, material of the present invention has a normalized drying power energy of from about 6,000 to about 16,000 ergs/g.

The unique combination of strength, absorptive ability and suppleness seen in the present absorbent material has significant advantages to a manufacturer of absorbent articles. Typically such a manufacturer purchases pulp and has to process that pulp on-line in their manufacturing plant as the final article (e.g., diaper, sanitary napkin) is being made. Such processing steps may include defibering of the pulp, adding superabsorbent and the like. In an on-line system, the rapidity with which such steps can be carried out is limited by the slowest of the various steps. An example of a pulp that requires such processing steps (e.g., defibering) is disclosed in U.S. Patent No. 5,262,005.

The need of the manufacturer to defiberize or otherwise process existing materials on-line means that the overall production process is substantially more complex. Further, the manufacturer must purchase, maintain and operate the equipment needed to carry out such processing steps. The overall production cost is thus increased.

An absorbent material of the present invention can be directly incorporated into a desired absorbent article without the need for such processing steps. The manufacturer of the absorbent article does not have to defiber or otherwise treat the materials of the present invention in any way other than shaping the material into

the desired shape. In this way, the manufacturer can speed up the assembly process and realize substantial savings in cost and time.

5 The material of this invention can be formed as a single layer of blended cellulosic fibers and superabsorbent material or air-laid as a plurality of layers or strata. In another embodiment, the material is formed as two layers. Each of the layers can contain cellulosic fibers and superabsorbent material although it is possible to limit the superabsorbent material to only one layer. A preferred material of the present invention is air-laid as three or four lamina or strata. Those strata include a bottom layer, one or two middle layers and a top layer. Preferred 10 embodiments of three and four layer material are set forth below. The superabsorbent material can be included in any or all of the layers. The concentration (weight percent) of superabsorbent material in each layer can vary as can the nature of the particular superabsorbent material. Five or more layer material is also contemplated by this invention.

15 An unexpected characteristic of the material of this invention is its ability to retain superabsorbent material when subjected to mechanical stress. In contrast to conventionally formed core materials, the material of the present invention retained over 85 percent by weight of its superabsorbent material content when subjected to 10 minutes of rigorous shaking (See, e.g., Example 4). Preferably, a material of this invention retains over 90 percent, preferably over 95 percent and, more preferably 20 over 99 percent of its superabsorbent material under these mechanical stresses.

Even where prepared as from multiple layers, the final thickness of the formed material is low. The thickness can vary from about 0.5 mm to about 2.5 mm. In a preferred embodiment, the thickness is from about 1.0 mm to about 2.0 25 mm and, more preferably from about 1.25 mm to about 1.75 mm.

One embodiment of an absorbent material of the present invention is particularly well suited for use in feminine hygiene products (See FIG.2). Such a material has a basis weight of from about 100 g/m² to about 250 g/m² and a density between about 0.25 g/cc and 0.5 g/cc. More preferably, the density is from about 30 0.3 g/cc to about 0.45 g/cc and, most preferably about 0.4 g/cc.

In one embodiment, a material for use in a feminine hygiene product is air-laid as three strata: a bottom layer of pulp (without superabsorbent) with a basis weight of about 25 g/m²; a middle layer with a basis weight of about 150 g/m² and which contains from about 10 g/m² to about 30 g/m² superabsorbent and from about 120 g/m² to about 140 g/m² pulp; and a top layer of pulp (without superabsorbent) with a basis weight of about 25 g/m². Relative to the total basis weight of the material, the level of superabsorbent ranges from about 5 to about 15 weight percent (g/m² of superabsorbent per g/m² material). Preferably, the level of superabsorbent is from about 7.5 weight percent to about 12.5 weight percent of the material. Most preferably, the material contains about 10 weight percent of superabsorbent. Thus, the middle layer of the material preferably contains from about 15 g/m² to about 25 g/m² superabsorbent and from about 125 g/m² to about 135 g/m² pulp and, more preferably about 20 g/m² superabsorbent and about 130 g/m² pulp. The middle layer containing pulp and superabsorbent can be laid down as a homogeneous blend or as a heterogeneous blend wherein the level of superabsorbent varies with proximity to the bottom layer.

In another embodiment, the material is air-laid as four strata. In this embodiment, the middle layer referred to above is replaced with two middle layers: a first middle layer adjacent the top layer and a second middle layer adjacent the bottom layer. Each of the first and second middle layers independently comprises from about 10 to about 30 g/m² superabsorbent and from about 40 g/m² to about 65 g/m² pulp. When it is desired to keep absorbed fluid away from the top of the feminine hygiene product (i.e., away from the surface of the article in closest proximity to the wearer) the amount of superabsorbent in the first and second middle layers is adjusted such that there is a higher level of superabsorbent in the second middle layer. The superabsorbent in the first and second middle layers can be the same or a different superabsorbent.

Another embodiment of an absorbent material of the present invention is particularly well suited for use in diapers and incontinence products (FIG. 3). Because such articles are expected to absorb and retain larger quantities of less viscous fluid than a feminine hygiene article, such a material is heavier and, thus,

has a preferred basis weight of from about 350 g/m² to about 450 g/m². The overall density of that material is between about 0.3 g/cc and 0.5 g/cc. More preferably, the overall density is from about 0.25 g/cc to about 0.45 g/cc and, most preferably about 0.4 g/cc.

5 In a manner similar to that described above, a material suitable for use in diapers can be air-laid as two, three or four strata. When three strata are used, a bottom layer has a basis weight of about 50 g/m²; a middle layer has a basis weight of about 300 g/m² and contains from about 40 g/m² to about 200 g/m² superabsorbent and from about 100 g/m² to about 260 g/m² pulp; and a top layer has
10 a basis weight of about 50 g/m². Preferably, the middle layer contains from about 70 g/m² to about 170 g/m² superabsorbent and from about 130 g/m² to about 230 g/m² pulp. Even more preferably, the middle layer contains about 80 g/m² superabsorbent and about 220 g/m² pulp or about 160 g/m² superabsorbent and about 140 g/m² pulp. The middle layer containing pulp and superabsorbent can be
15 laid down as a homogeneous blend or as a heterogeneous blend wherein the level of superabsorbent varies with proximity to the bottom layer.

In a four strata embodiment, the middle layer is replaced with a first and second middle layer oriented as set forth above. Each of the first and second middle layers independently contains from about 20 g/m² to about 100 g/m² superabsorbent
20 and from about 50 g/m² to about 130 g/m² pulp. In a preferred embodiment, the second middle layer has a higher level of superabsorbent than the first middle layer. In this way, the formed absorbent material has a tendency to keep absorbed fluid away from the body surface of the wearer of the article. The superabsorbent in the first and second middle layers can be the same or a different material.

25 An absorbent material for the present invention can be incorporated into an absorbent as a single or multiple-ply structure. Means of forming multiple-ply structures using folding are well known in the art. By way of example, a person skilled in art can "C", "G" or "Z" fold the absorbent material of the present invention prior to incorporating it into an absorbent article.

30 The following Examples illustrate preferred embodiments of the present invention and are not limiting of the specification and claims in any way.

Example 1:

A 400 g/m² absorbent material was produced in accordance with the above procedure using cold caustic extracted pulp. Overall composition of the web was 60% pulp and 40% Stockhausen T5318 superabsorbent material. The Gurley stiffness values of the absorbent material as well as absorbent cores from commercially available diapers were measured using a Gurley Stiffness Tester (Model No. 4171E), manufactured by Gurley Precision Instruments of Troy, NY. The instrument measures the externally applied moment required to produce a given deflection of a test strip of specific dimensions fixed at one end and having a concentrated load applied to the other end. Those commercial core materials were densified to achieve a range of densities comparable to the material of the present invention. The results are obtained in "Gurley Stiffness" values in units of milligrams. It should be noted that the higher the stiffness of the material, the less flexible and hence the less soft it is. Table 1 presents results of this test.

Table 1**Effect of Density On Gurley Stiffness**

Absorbent Mat'l.	Stiffness (mg)	1021	1175	1575
	Density (g/cc)	0.34	0.43	0.5
	Ratio (stiffn/den)	3303	2732	3150
Huggies®	Stiffness (mg)	1313	2450	3775
Ultratrim	Density (g/cc)	0.31	0.4	0.51
Med. Diaper	Ratio (stiffn/den)	4235	6125	7401
Pampers®	Stiffness (mg)	1638	2350	4400
Baby Dry	Density (g/cc)	0.3	0.42	0.51
Stretch Med. Diaper	Ratio (stiffn/den)	5460	5595	8627

Example 2:

Absorbent materials of the present invention were made with basis weights of 400 g/m² and 600 g/m², containing 20 and 40 weight percent superabsorbent material, respectively. Wicking properties of the material and a core from a Huggies® diaper were measured using GATS system manufactured by M/K Systems of Danvers, MA. Fig. 4 illustrates set up of the test. A 45° wicking test cell is attached to the absorption measurement device. The test cell essentially consists of a circular fluid supply unit for the test sample and 45° ramps. The fluid supply unit has a rectangular trough and liquid level is maintained at the constant height by the measuring unit. The test sample having dimension of 1" x 12" was prepared. The sample was marked every inch along the length of the sample. The sample was then placed on the ramp of the test cell ensuring that one of the edges of the sample dips into the trough. The test was conducted for thirty minutes. Sample was removed after the specified period and cut along the marked distances. The cut pieces were placed into pre-weighed aluminum weighing dishes. The weighing dish containing wet samples were weighed again and then oven dried to a constant weight. By conducting a proper mass balance on the data, absorbency of the sample was determined at every inch. The following Table 2 presents results of the test:

Table 2
Absorbency (g/g)

	Wicked Distance (in)	400/20 Mat'l.	600/40 Mat'l.	Huggies® Diaper Core
25	2	19.4	18.9	19.4
	3	16.6	17.2	16.3
	4	15.3	15	12.4
30	5	12.2	11.9	4.9
	6	7.7	7.9	0.3
	7	1.7	1.0	
	8	0.2		

The data show that the absorbent web of the present invention has a capability of transporting large amounts of fluid very rapidly from the liquid source.

Additional studies were performed using a 400 g/m² basis weight, 0.40 g/cc density, 40 weight percent superabsorbent material of the present invention (C11, C12 and C13) and the absorbent cores from commercially available Huggies® and Pampers® diapers; and commercial roll goods from Merfin and Concert. The results of those studies are summarized below in Table 3.

Table 3

Distance (in)	C11 (15% SAP)	C12 (28% SAP)	C13 (42% SAP)	Huggies® (36% SAP)	Pampers® (43% SAP)	Merfin (40% SAP)	Concert (30% SAP)
2	15.3*	19.3	22.3	19.3	24.6	17.9	11.6
3	13.9	17.1	20.6	17.7	19.6	15.5	11.2
4	12.7	15.6	19.1	16.4	14.6	9.2	8.5
5	11.6	13.8	17.4	13.9	10.6	1.06	5.7
6	10.4	12.2	14.4	10.5	5.5	0.16	0.82
7	9.1	9.9	8.3	4.4	1.4	0.16	0.04
8	7.7	6.7	0.75	0.50	0.1	0.15	0.01
9	5.8	1.4	0.10	0.00	0.00	0.00	0.02
10	2.4	0.07	0.08	0.00	0.00	0.00	0.00
11	0.07	0.07	0.10	0.00	0.00	0.00	0.00
12	0.07	0.07	0.09	0.00	0.00	0.00	0.00

* g/g

The results show the superior wicking ability of an absorbent material of the present invention when compared to commercially available material. It can be seen that material of the present invention wicked substantial amounts of fluid at a distance greater than 7 inches. In contrast none of the commercially available material distributed significant amounts of fluid beyond that distance.

Example 3:

A series of samples was evaluated for the integrity of the absorbent core in a range of material density from about 0.20 g/cc to about 0.50 g/cc. The test is

performed on an Instron Universal Testing Machine. Essentially, the test measures the load required to pierce through the test sample, as described in the PFI Method of 1981. A test sample having dimensions of 50x50 mm is clamped on the Instron with a suitable fastening device. A 20 mm diameter piston traveling at the rate of 50 mm/min punctures the stationary sample. The force required to puncture the sample is measured. The following Table 4 presents results of the test.

Table 4**Density vs. Core Integrity**

10	Absorbent Mat'l.	Pad Integrity (N)	12.4	16.3	15.8
		Density (g/cc)	0.234	0.326	0.433
		Ratio (integ/den)	53	50	36.5
15	Huggies® Ultra-trim Med. Diaper	Pad Integrity (N)	5.11	7.6	8.24
		Density (g/cc)	0.254	0.328	0.455
		Ratio (integ/den)	20.4	23.0	18.2
20	Pampers® Baby Dry Stretch Diaper	Pad Integrity (N)	3.9	6.6	9.37
		Density (g/cc)	0.26	0.38	0.42
		Ratio (integ/den)	15.0	17.4	22.3

The above data clearly indicate the absorbent material produced by the above invention is stronger than the conventional absorbent cores in the commercial diapers.

Example 4:

An amount of loosely held superabsorbent material in various absorbent materials was determined by shaking the material in a Ro-Tap Sieve Shaker manufactured by W. S. Tyler Co., Cleveland OH. Absorbent cores from commercial diapers, Huggies® Ultratrim and Pampers® Baby-dry Stretch, containing approximately 40% by weight of superabsorbent material granules were carefully removed and placed in a 28-mesh (Tyler series) sieve. Additional sieves of 35-mesh and 150-mesh were attached to the first sieve forming a column of increasingly fine sieves. The column of sieves was capped on either end to prevent the loss of fiber and/or superabsorbent material. The sieve column was placed in the shaker and agitated for 10 minutes. The amount of superabsorbent material

granules shaken loose from the absorbent cores, "free superabsorbent material", was determined by combining the residue contained in each of the sieves and separating the cellulosic fiber from the superabsorbent material. Comparative data for a present absorbent material containing 40% superabsorbent material were obtained in a similar fashion. The material was formed as in Example 1.

Data in Table 5 show that the present absorbent materials retained 100% of the superabsorbent material granules while the commercial cores from the Huggies® and Pampers® products lost approximately 16.6% and 29.5% by weight of the total superabsorbent material contained in the core.

Table 5

Determination of Free superabsorbent material in Absorbent Cores

	Product	Total Core Weight	Superabsorbent Weight	Free Superabsorbent Material	% Free Superabsorbent Material
15	Huggies® Ultratrim	22.63 g	9.05 g	1.51 g	16.6
20	Pampers® Baby-Dry Stretch	20.10 g	8.04 g	2.37 g	29.5
	Absorbent Material	20.45 g	8.18 g	0.00 g	0.0

Example 5:

Absorbent material produced as in Example 1 was calendered with smooth and engraved (patterned) rolls to achieve desired density. Absorption capacity of the material against various applied pressures was measured by placing a known weight on top of the absorbent material, the known weight representing a specific pressure against the absorbent material, then contacting the absorbent material with a standard (0.9%) saline solution and allowing the material to absorb fluid until an equilibrium condition is attained. The following Table 6 presents results of the test:

Table 6
Effect Of Embossing On Absorbent Capacity

	Calender Type	Basis Weight Density		Absorption Against Load	
		g/m^2	g/cm^3	(g/m^2) 0.3 psi	0.7 psi
10	Engraved	352	0.36	5430	4394
	Smooth	405	0.35	5871	4666
	Engraved	546	0.34	7912	6364
	Smooth	596	0.35	8169	6518

15 It is evident from the test data that absorbency of calendered material with the pattern rolls is lower than the material calendered by the smooth rolls. The pattern roll essentially embosses the material. The lower absorbency may be due to damage caused to the superabsorbent material particles or to the introduction of very highly densified areas into the material as a result of the embossing. Damage to superabsorbent material granules and creation of super-densified zones in the absorbent material can have a negative impact on absorbent capacity. Therefore, it is preferred to calender the material with a smooth roll.

Example 6:

25 45 Degree wicking absorption was determined using the procedures of Example 2. The following groups of samples were tested: (a) absorptive material of the present invention with a basis weight of about 400 g/m^2 , a density of about 0.4 g/cc , and varying superabsorbent material contents of about 15 weight percent (Sample C11), 28 weight percent (C12), 39 weight percent (C1) or 42 weight percent (C13); (b) thermal bonded air-laid fluff obtained from Concert (Concert 500, 280, 130) or Merfin (44500); the absorbent core removed from a Huggies® Diaper; and the absorbent core removed from a Pampers® diaper. Samples C11, C12 and C13 were made using 100 percent cold caustic treated fibers. Sample C1 was made using a blend of 50 percent cold caustic treated fibers and 50 percent non-

cold caustic treated fibers. For each sample, the amount of fluid absorbed per gram of sample was plotted against distance from the origin (source of fluid). A representative plot is shown in FIG. 5. The area under the curve was calculated using the following formula:

$$[(y_1)(x_2 - x_1) + 0.5(y_2 - y_1)(x_2 - x_1) + (y_2)(x_3 - x_2) + 0.5(y_3 - y_2)(x_3 - x_2) + \dots + (y_n)(x_n - x_{n-1}) + 0.5(y_n - y_{n-1})(x_n - x_{n-1})], \text{ where } X_i \text{ is distance at the } i^{\text{th}} \text{ inch and } Y_i \text{ is absorbency at the } i^{\text{th}} \text{ inch.}$$

This area was then multiplied by the gravitational constant (981 cm/s²) and the sine of 45° to result in the work value of ergs/g. The derived energy value was normalized for superabsorbent material by dividing by percent superabsorbent material (%SAP) content. The results of these studies are summarized below in Table 7.

Table 7				
Sample	% SAP	Total Wicking Energy (ergs/g)	Normalized Wicking Energy (ergs/g)	Density (g/cc)
C 1	39	161,299	4,136	0.38
C 11	15	143,295	9,553	0.36
C 12	28	152,509	5,447	0.36
C 13	42	162,200	3,862	0.38
Concert 500	45	93,016	2,067	0.12
Concert 280	30	67,216	2,241	0.17
Concert 130	18	56,219	3,123	0.13
Merfin 44500	40	62,094	1,552	0.17
Huggies®	36	133,889	3,719	0.15
Pampers®	42	112,870	2,625	0.12

The data show that material of the present invention demonstrated superior wicking power when compared to the other materials.

Example 7: Normalized Drying Power Energy

Various absorptive materials of the present invention, as well as commercially available absorptive materials (See Example 6 above), were

examined to determine their ability to absorb fluids against a negative hydrostatic pressure gradient. The means used to determine this absorptive ability of the material (referred to herein as normalized drying power energy) were obtained using the well known drying power test (reference Burgeni et al., *Textile Research Journal*, 37 1967 362). Absorbency is measured under varying hydrostatic pressure heads (tension). Drying power energy is derived from the absorbency data.

Absorbency of the sample is measured at various negative hydrostatic pressures, i.e., negative hydrostatic heads. The negative hydrostatic pressure exerts a suction force on the sample. The absorbent material needs to have enough positive force to overcome the negative suction force in order to absorb fluid. The positive force results from the capillary pressure of the fiber matrix and osmotic pressure of the superabsorbent polymer. As the absorbent material picks up fluid, the positive pressure decreases. A point is reached when the positive force necessary to counter-balance the suction force is insufficient. This point is referred to as equilibrium absorbency and represents the cessation of absorption. The hydrostatic pressure is systematically lowered in 5 cm increments from 35 cm to 1 cm of water, and the equilibrium absorbency at each hydrostatic tension value is measured. At a hydrostatic tension value of about 1 cm of water, the fiber network is completely saturated with the test fluid and the superabsorbent material polymer is fully hydrated. This point represents maximum absorption.

A schematic illustration of an instrument used to obtain measurements for this characterization is shown in FIG. 6. As can be seen from FIG. 6, the instrument comprises a fluid source as well as an adjustable sample compartment. The fluid source comprises a constant-level fluid reservoir in conjunction with a supply reservoir. The entire fluid reservoir component is placed on a balance to allow for determination of the mass of the fluid lost or gained by the fluid reservoir. The fluid source is connected via a tube to the adjustable sample compartment. The adjustable multiport compartment (available from M-K Systems of Danvers, MA) comprises a solid support on which is placed a filter paper (Whatman #5) and a sample of absorbent material. The solid support mechanism together with the filter and sample are attached to a device which allows for raising and lowering of the

sample height relative to the height of the fluid in the fluid reservoir. When the level of the sample and the sample compartment is the same as the level of the constant level fluid reservoir, there is 0 hydrostatic pressure head applied to the sample. As the sample level is raised above the level of fluid in the constant level reservoir, a negative hydrostatic pressure head is applied to the sample. The magnitude of the hydrostatic pressure head is equal to the difference in height between the sample and the fluid reservoir as measured in centimeters.

The various samples of absorptive material were placed in the instrument and fluid absorption measured over a range of hydrostatic pressures. The amount of fluid absorbed at each pressure (normalized for sample dry weight) was plotted against hydrostatic pressure. A representative plot is shown in FIG. 7. The area under the curve from point A to point Y is integrated. Drying power energy (ergs/g) is defined as this area. Normalized drying power energy is defined as the drying power energy value divided by the % superabsorbent material in the sample. The results of these studies are summarized below in Table 8.

Table 8

Sample	% SAP	Total Drying Power Energy (ergs/g)	Normalized Drying Power Energy (ergs/g)	Density (g/cc)
C 1	39	283,622	7,272	0.38
C 11	15	241,163	16,078	0.36
C 12	28	276,103	9,861	0.36
C 13	42	356,667	8,492	0.38
Concert 500	45	105,345	2,341	0.12
Concert 280	30	162,303	5,410	0.17
Concert 130	18	141,592	7,866	0.13
Merfin 44500	40	172,099	4,302	0.17
Huggies®	36	161,686	4,491	0.15
Pampers®	42	95,972	2,285	0.12

The data in Table 8 show that a material of the present invention has superior normalized drying power energy when compared to commercially available materials of comparable density.

Example 8:

The samples used in Examples 6 and 7 were analyzed to determine their suppleness. Gurley stiffness measurements were obtained using the procedures of Example 1. The data from these studies are summarized below in Table 9.

Table 9
Suppleness

<u>Sample</u>	<u>% SAP</u>	<u>Suppleness</u> (g ⁻¹)	<u>Density</u> (g/cc)
C1	39	0.74	0.38
C11	15	0.792	0.36
C12	28	0.898	0.36
C13	42	1.235	0.38
Concert 500	45	0.612	0.12
Concert 280	30	1.429	0.17
Merfin 44500	40	0.374	0.17
Huggies®	36	0.890	0.15
Pampers®	42	0.727	0.12

The data in Table 9 show that a high density material of the present invention has a suppleness comparable to that of low density commercially available samples.

Example 9:

Absorbent cores were carefully removed from Huggies® and Pampers® commercial diapers. The core was cut 35.88 cm long and 9.53 cm wide (14 1/8 in x 3 3/4 in). The absorbent cores were placed on 1.0 mil polyethylene and covered with a nonwoven cover (PGI Thermal Bonded Nonwoven Cover). In a similar fashion, a material of the present invention (basis wt. of 400 g/m², density of 0.40 g/cc, 40 weight percent superabsorbent material, a blend of cold caustic extracted and non-cold caustic extracted pulp) was placed on polyethylene and covered. All samples were tested for fluid acquisition and rewet using standard procedures well known in

the art. These tests measure the rate of absorption of multiple fluid insults to an absorbent product or material and the amount of fluid that is rewet under 0.5 psi load. This method is suitable for all types of absorbent material, especially those intended for urine application.

5 Briefly, a fixed amount of saline solution is absorbed by an absorbent product or material. The absorption is recorded and a 30 minute absorption and wicking period follows. A filter paper and a 0.5 psi load is then applied to the test sample for 2 minutes. The fluid acquisition time and volume of rewet are recorded. This absorption and rewet process is repeated 3 times. Each value is reported along
10 with the average and the standard deviation. This test measures rate of absorbency and absorption capacity. This test is performed in triplicate to verify results. For the present studies, 50 ml of saline were used as the fluid load. In addition to determining the acquisition times and rewet masses after the third insult, the wicking distance for each sample was calculated. The results of these studies are
15 summarized below in Table 10.

Table 10
Acquisition and Rewet Test

Sample	Core Weight (g)	Percent SAP	Third		Weight Difference %	Wicking Length (cm)
			Acquisition Time (s)	Rewet (g)		
Pampers®	19.504	43 %	86	2.246	25	15.24
Huggies®	21.023	36 %	82	1.559	35	19.05
Absorb. Mat'l	15.602	37 %	47	0.63	—	27.31

It can be seen from the data in Table 10 that, despite a reduced sample weight, the material of the present invention showed a greater wicking length and
30 a lower rewet value than commercially available materials.

Example 11: X-ray Diffraction Studies

X-ray diffraction (XRD) is a technique used for determining atomic and molecular structure of crystalline materials. For XRD analysis, the sample is bombarded with an x-ray beam (typically CuK_α x-rays). Crystal planes and individual atoms within the crystal plane scatter and diffract the x-ray beam. Every crystalline substance produces a unique diffraction pattern that is essentially a "fingerprint" of the compound's crystalline structure. Diffraction patterns are reproduced either on a film strip or as a computerized spectrum. Comparisons are made to reference patterns from databases such as JCPDS (Joint Committee on Powder Diffraction Standards) files to identify the compound.

The relative crystallinity of four cellulosic fibers used in absorbent materials was determined using XRD analysis. The four samples were prepared by the kraft, method from Southern Pine wood. All samples were compressed to a density of 0.35 to 0.40 g/cc and a basis weight of 350 to 450 g/m². A control sample (A) was neither cold caustic extracted nor flash dried. A second sample (B) was prepared from pulp that was flash dried as described hereinbefore. A third sample (C) was prepared from pulp that was extracted with a cold, 13 weight percent solution of NaOH. A fourth sample (D) was prepared from pulp extracted with a cold, 16 weight percent NaOH.

A 25 mm square of material was cut from each sample and the corners rounded to produce a circular sample approximately 25 mm in diameter. Each circular sample was then mounted on an aluminum disc using double-side tape. The aluminum disc was approximately 25 mm in diameter and 1 mm thick. The disc-sample assembly was then placed into a square holder and held in place by a retaining ring. Sample height adjustment necessary to maintain the correct source to sample geometry was adjusted using the retaining ring. Each sample was then x-rayed from 2° to 40° 2θ using copper radiation, an accelerating voltage of 45 KeV, a beam current of 40 mA, a step size of 0.05° 2θ and a data acquisition time of 2.0 seconds per step. The samples were also rotated during analysis. X-Ray diffractograms were obtained from each sample. FIG. 8 is a representative x-ray diffractogram obtained using Example B.

The relative percent crystallinity of the samples was calculated from the data in each diffractogram. The principles involved in the calculation are illustrated in FIG. 8. As a first step in the calculation, a line x-y was drawn tangent to the baseline at 7° and 32°. Second, a curve is constructed tangent to the peak minima. The area under that curve (A_{NCP}) is designated as the Non-Crystalline Peak area. The area above that curve (A_{CP}) is designated as the Crystalline Peak area. The relative crystallinity is then calculated as [Crystalline Peak area (A_{CP}) ÷ Total Area ($A_{CP} + A_{NCP}$)] x 100. The values for A_{CP} and A_{NCP} can be determined using any number of means well known in the art.

The results of the above studies are shown in Fig. 9 and summarized in Table 11 below.

Table 11

Sample	Relative Crystallinity %
A	68-69
B	52-54
C	46-47
D	36-37

It can be seen from the data in Table 11 that both cold caustic extraction and flash drying substantially decrease the relative crystallinity of the pulp.